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BIAXIALLY ORIENTED POLYESTER FILM

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(57). (Abstract).

(Subject).

To put forward a polyester film useful as base film of magnetic recording medium having excellent smoothness and sliding properties.

(Construction).

A biaxially orientated polyester film of the kind wherein the film mainly comprised polyester, characterised in that aggregable inorganic fine particle (particle A) having primary particle size of 30 nm or less is contained in an amount of 6.0 X 10 [power 17] or less per 1 kg of said film, and particle

(particle B) having average particle diameter of 400 nm or less and also having an average particle diameter larger than the length of aggregate consisting of particle A in the film in the film thickness direction is contained so as to be 0.1-2.0 cm3 per 1 kg of said film.

Patent Claims

Claim 1

A biaxially orientated polyester film of the kind wherein the film mainly comprised polyester, characterised in that aggregable inorganic fine particle (particle A) having primary particle size of 30 nm or less is contained in an amount of 6.0 X 10 [power 17] or less per 1 kg of said film, and particle (particle B) having average particle diameter of 400 nm or less and also having an average particle diameter larger than the length of aggregate consisting of particle A in the film in the film thickness direction is contained so as to be 0.1-2.0 cm3 per 1 kg of said film.

Detailed Description of the Invention

(0001)

(Sphere of Application in Industry).

This invention relates to a film consisting of polyester, suitable for base film for magnetic recording medium.

(0002)

(Technology of the Prior Art and Problems to be Overcome by this Invention)

Recently, high density formation of magnetic recording density is encouraged, and therefore, improvement of electromagnetic conversion characteristics is required. As a direction to improve the electromagnetic conversion characteristics, in general, a process to flatten the magnetic recording layer is known, and the flattening of the surface of polyester film which is used as base film is required for this purpose. The formation of flat polyester film can be easily achieved, however, unless the film surface has appropriate surface roughness, the sliding properties and workability are remarkably decreased, moreover, the abrasion resistance falls, too. Accordingly, generally, particles are added in the polymer, film surface projections are formed, and the contact points on the film is decreased in effect. In the prior art, various techniques have been suggested in order to establish both these sliding properties and flatness. For example, there is a method and the like

wherein sliding properties are imparted by minute projections, and for the purpose of improving workability and winding properties, projections relatively greater than aforesaid projections are established in small number. However, accompanied with the high density formation of magnetic recording density, the large projections formed for improving the winding property cause drop out and lowering of electromagnetic conversion characteristic, and improvement is required.

(0003)

(Means to Overcome these Problems).

These inventors carried out assiduous investigations into these issues, as a result, discovered that particle groups of specific particle size are caused to be present in the film in specific quantities, thereby both the sliding property and flatness was able to be established, and sliding property and winding characteristic were imparted without lowing the electromagnetic conversion characteristic. This invention was completed based on this discovery.

(0004)

In other words, the gist of this invention is a biaxially orientated polyester film of the kind wherein the film mainly comprised polyester, characterised in that aggregable inorganic fine particle (particle A) having primary particle size of 30 nm or less is contained in an amount of 6.0 X 10 [power 17] or less per 1 kg of said film, and particle (particle B) having average particle diameter of 400 nm or less and also having an average particle diameter larger than the length of aggregate consisting of particle A in the film in the film thickness direction is contained so as to be 0.1-2.0 cm3 per 1 kg of said film.

(0005)

Hereinafter, this invention will be described in greater detail. The polyester in this invention is a polyester mainly comprising aromatic dicarboxylic acid and aliphatic glycol, and it may be polyethylene terephthalate which is widely used as base film for magnetic recording medium, however, polyethylenenaphthalate is more preferred. Polyethylenenaphthalate has higher modulus of elasticity compared to polyethylene terephthalate and deformation of contact site is small, therefore, it is known to be more easily slid under the same composition of added particles. The polyethylenenaphthalate described here is a polyester containing at least 80 pts. naphthalene-2,6-

dicarboxylic acid component as acid component and at least 80 pts. ethylene glycol component as glycol component, and species wherein in addition to these components, acid components such as isophthalic acid, phthalic acid, terephthalic acid, adipic acid, sebacic acid or the like and glycol component such as propylene glycol, butanediol, 1,4-cyclohexane dimethanol, neopentyl glycol and the like are copolymerized, or species containing a hydroxy acid such as p-hydroxybenzoic acid as monomer component may be used.

(0006)

Moreover, it may be species which contains in accordance with requirements additives such as stabilizer, colorant, antioxidant, antifoaming agent, antistatic agent and the like. In this invention, as particle A, aggregable inorganic fine particles having primary particle size of 30 nm or less for example silicon oxide, alumina, carbon black and the like are contained in an amount of 6.0 x 10 [power 17] or less per 1 kg film. Particle A is contained, for example, by processes such as slurry addition during polymerization, kneading into polymerized polymer chip or the like. Wherein, the aggregable inorganic fine particle refers to in organic particles wherein the quantity of particles present singularly as primary particle in the film after molding is less than 30 % of the total, plurality of particles form aggregates, and behave as aggregate to some extent under strong stress such as drawing and the like, however, the aggregates are deformed corresponding to the deformation of film, and only a smaller internal void compared to the secondary particle diameter before deformation is formed. When the number of particle added is greater than 6.0 x 10 [power 17] or less per 1 kg film, the film surface roughness becomes too high and it is not preferred.

(0007)

As particle B used in this invention, for example, silicon oxide, calcium carbonate, an organic particle formed by crosslinking monomer component, titanium oxide and the like are proposed, the average particle diameter of particle B is 400 nm or less, preferably 300 nm or less, more preferably 200 nm or less and also is greater than the length of aggregate consisting of particle A in the film in the film thickness direction. The particle having such average particle diameter is contained by slurry addition during polymerization, kneading into polymerized polymer chip or the like so that the total particle volume per film 1 kg comprises 0.1-2.0 cm3. When the total particle volume is less than 0.1

cm3, the improvement effect of workability is small, and when the total particle volume is greater than 2.0 cm3, surface is not flat and it is not preferred.

(0008)

Although, a flatness and sliding properties are imparted to some extent with single particle system consisting of each particle A or B, the particle system consisting of particle groups having different particle sizes as in this invention has superior travelling properties and winding characteristic. A film which is a biaxially orientated polyethylenenaphthalate film containing aforesaid particle groups, wherein the center line average roughness of 8 nm or less, preferably 7 nm or less, more preferably 6 nm, and the with frictional coefficient is 0.55 or less, is a film having a high degree of flatness and easy sliding property. The process for the production of film of this invention is not limited in particular, and a coated layer may be preferably established during the film production step or after the film production corresponding to applications thereof.

(0009)

Examples

Hereinafter, Examples are nominated, and this invention is described in further detail, however, will not be restricted by following Examples so long as the gist of this invention is not exceeded. Moreover evaluation process in Examples are as follows. Moreover, "pts." in Examples and Comparative Examples denotes "parts by weight".

(1) Center line average roughness, Ra

It was performed by a process in accordance with JIS BO601-1976. Roughness meter (SE-3F) made by Kosaka Laboratories KK. was used for the measurement. The centre line average roughness was measured under conditions comprising probe diameter of 2 μm, probe load of 30 mg, cut-off value of 0.08 mm and measurement length of 0.8 mm. This was carried out at 12 measurement points, from these, the maximum value and the minimum value were respectively disregarded, and the Ra was determined by the average value of 10 points.

(0010)

(2) Sliding property μd

Two sheets of films excised in 15 mm wide and 150 mm long were superimposed on a flat glass plate, a rubber plate was placed on top, a load was further placed on top, the films were caused to slide each other with contact pressure of two films of 2 g-mass/cm2 at 20 mm/min, and the frictional force was measured. The frictional coefficient when the films were slid by 5 mm was determined as dynamic friction coefficient μd.

(3) Winding property

When wound up in a roll, the appearance of the surface of the film roll and the edges are evaluated as follows:

O: Little creases or grainy defects are present on the roll surface, and the edges are even.

Δ: Little creases are present on the roll surface, however, grainy defects are generated slightly, the edges are slightly uneven.

X: Creases and grainy defects are generated on the roll surface, or the edges are extremely uneven.

(0011)

(4) Particle number

The particle number of the film per 1 kg film was calculated by assigning the primary particle diameter d [mm] measured by transmission electron microscopy, the specific gravity, rho of substance constituting the particle [g/cm3], added pts.wt., c [%] into the following equation.

Particle number = (6 x c) x 10 [power 13] / (pi x rho x d [power 3])

(5) Total particle volume

The total particle volume per 1 kg film was calculated by assigning the specific gravity, rho of substance constituting the particle [g/cm3], added pts.wt., c [%] into the following equation.

Total particle volume per 1 kg film = (10 x c) / (rho) [cm3]

(6) The length of aggregate particles in the film thickness direction.

The film cross section was photographed by transmission electron microscopy, the maximum diameter obtained by fitting two lines parallel to the thickness direction of the film is determined, a rectangle having such maximum diameter and also having the same area as aforesaid aggregate is formed, and the length of the other side of the rectangle was regarded as the length of the aggregate in the thickness direction of the film. The length was determined as average of 100 aggregates.

(0012)

Example 1

Naphthalene-2,6-dimethyl dicarboxylate ester 100 pts., ethylene glycol 60 pts. and calcium acetate monohydrate 0.1 pts. were charged to a reactor, and transesterification reaction was carried out. In other words, the start of reaction temperature was set at 180°C, the reaction temperature was gradually increased with distillation of methanol, and, after 4 hours, it was warmed to 230°C, and transesterification reaction was substantially completed. Next, phosphoric acid 0.04 pts. and ethylene glycol slurry containing spherical silicon oxide having particle size of 0.19 μm as particle B were added, thereafter, antimony trioxide 0.04 pts. was added, and polycondensation was carried out in accordance with conventional procedures. In other words, the temperature was gradually raised, the pressure was gradually reduced, and after 2 hours the temperature was set at 290°C, pressure was set at 0.3 mmHg, and the polyethylenenaphthalate containing silicon oxide 0.1 pts. was obtained.

(0013)

When four hours had elapsed from the start of reaction, the reaction was stopped, and polyester was discharged under pressurised nitrogen. This molten polymer was transferred to an extruder without further treatment, was filtered with a filter, thereafter, was extruded in strands and cut into chip-form. As particle A, fine silica particle aggregate having particle size measured by sedimentation method of 0.15 μm was kneaded into the obtained chip in an amount of 0.1 wt.% using biaxial kneading machine, and polyester raw material having limiting viscosity of 0.68 was obtained.

(0014)

Next, the obtained polyester was dried, melt-extruded, and amorphous sheet was made using electrostatic application cooling method. The obtained amorphous sheet was drawn by 4.0 times in longitudinal direction at 130°C, and next, an aqueous coating agent comprising 95 pts. water soluble polyester containing terephthalic acid 92 mol.%, sodium sulfoisophthalic acid 8 mol.%, ethylene glycol 75 mol.% and diethylene glycol 25 mol.%, 5 pts. silica sol having average particle diameter of 0.03 μm and 1900 pts. water was applied to one side, and drawing was carried out by 4.0 times at 137°C in cross direction. Furthermore, drawing was carried out by 1.8 times in longitudinal direction at 140°C, thereafter drawing was carried out by 1.1 times in cross direction at 200°C, and while tentering, heat-setting was carried out at 220°C, and the sheet was wound up while relaxing by 4 % in longitudinal and cross directions during cooling. The discharge quantity was adjusted so that the film thickness of the final film became 6 μm.

(0015)

Example 2

A film was obtained in the same way as in Example 1, except that as particle B added during polymerization, 0.3 pts. of spherical particle comprising crosslinking polymer having particle size of 0.10 μm with small particle size distribution was added and the particle A to be knead after polymerization was changed to aluminium oxide in an amount of 0.3 pts..

Comparative Example 1

A film was obtained in the same way as in Example 1, except that particle B was not added.

Comparative Example 2

A film was obtained in the same way as in Example 1, except that as particle B added during polymerization, 0.02 pts. of spherical particle comprising crosslinking polymer having particle size of 0.64 μm with small particle size distribution was added.

(0016)

Comparative Example 3

A film was obtained in the same way as in Example 1, except that spherical silicon oxide having particle size of 0.31 μm and 0.64 μm was added in an amount of 0.09 pts. and 0.02 pts. respectively

during polymerization, and particles were not kneaded after poymerization. the results obtained in above are collated and shown in the following Table 1 and Table 2.

(0017)

(Table 1).

	Particle A	Particle size μm	Length of aggregate μm	Particle number per 1 kg film	Particle B	Particle size μm	Total particle volume cm3/1 kg
Example 1	SiO2	0.015	0.10	2.57×10^{17}	SiO2	0.19	0.15
Example 2	Al2O3	0.013	0.05	2.89×10^{17}	organic particle	0.07	0.87
Comp. Ex. 1	SiO2	0.015	0.10	2.57×10^{17}	-	-	-
Comp. Ex. 2	SiO2	0.015	0.10	2.57×10^{17}	organic particle	0.64	0.17
Comp. Ex. 3	SiO2	0.31	0.10	2.88×10^{17}	SiO2	0.64	0.1

(0018)

(Table 2)

	Ra (nm)	μd	Winding property
Example 1	5	0.42	O
Example 2	6	0.52	O
Comp. Ex. 1	5	0.61	X
Comp. Ex. 2	10	0.45	O
Comp. Ex. 3	9	0.39	O

(0019)

(Advantages Afforded by this Invention)

The polyester film of this invention has flatness and sliding properties, and the commercial value thereof is high.

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㊹考案の名称　タフテツドカーペット

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㊼実用新案登録請求の範囲

1　基布として繊維シートの表面または裏面に合繊ウエブを40～200g／㎡展綿してニードルパンチングで一体化したものを用い、この基布に所望のパイル糸を刺し込み、ついで基布裏面に適宜の接着材を塗布・乾燥して比較的短いパイルを形成し、このカーペット長尺物を一定サイズに切断した後に、ミシンフック機で別のパイル糸を刺し込んでシヤギーボーダー加工し、この刺し込み部分における基布裏面にさらに接着剤を塗布・乾燥して比較的長いパイルを形成することを特徴とするタフテツドカーペット。

2　繊維シート上に展綿する合繊ウエブはポリエステル繊維製である登録請求の範囲第1項に記載のカーペット。

考案の詳細な説明

この考案は、台地が薄くても機械的性質がすぐれたタフテツドカーペットに関する。

タフテツドカーペットを製造するには、タフト機によつてジユートなどの第1基布にパイル糸を刺し通し、該基布の裏面から接着剤を塗布してパイルが抜けないように固着するのが普通である。このようなノーマルバツキング法では、一般に台地が薄すぎてしつかりしないので、さらに第2基布としてジユート布や亀甲紗を裏貼りしたり、フオームラバーバツキングなどを行なうことが多い。この結果、カーペットのクツシヨン性を付与

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し、さらに断熱効果、吸音効果を増すうえで好ましい。しかしながら、台地が厚くなつて断熱効果が増すと、カーペットの下方に面状ヒータを設置して電熱カーペットまたはそのカバー材として用いる場合には、ヒーターからの伝熱性が低下するので好ましくない。またラグカーペットとして用いる場合には、一定の機械的性質を保持させながら運搬や保管のために台地を薄くて軽くすることは困難である。

この考案は、タフテツドカーペットの台地に関する前記の問題を改善するために提案されたものである。したがつてこの考案の目的は、繊維シートに合繊ウエブを一体化させた基布を用いることにより、台地が薄くても機械的性質がすぐれたタフテツドカーペットを提供することである。この考案のように台地を薄くできると、電熱カーペットやそのカバー材として用いる際に面状ヒータからの発熱を上方へ良く伝え、ラグカーペットとして用いる際には運搬や保管の点でも有利である。

この考案を図面に基いて説明すると、第1図および第2図にこの考案に係るタフテツドカーペット1をそれぞれ例示する。基布2を構成するために、繊維シート3の裏面（第1図）または表面（第2図）に合繊ウエブ4を展綿し、両者をニードルパンチングで一体化させる。繊維シート3としては、ジユートや綿の綾織布、ポリプロピレンのスリツトヤーンの平織布、ポリプロピレンやポ

リエステルのスパンボンド織布などが例示できる。また合繊ウエブ4は、ポリエステル、ポリプロピレンなどから製造し、その展綿量は40～200g／㎡であると好ましい。この際に、展綿量が40g／㎡未満であると基布2の機械的強度を増加させることが十分でなく、200g／㎡を超えると基布2が厚くなりすぎてタフテイングにおける機械的負荷が大きくなり、かつ製造コストの面でも不利になつてくる。合繊ウエブ4の位置は、繊維シート3の表面でも裏面でもよいが、基布2におけるパイルの抜糸強度をより高くするには繊維シート3の裏面に合繊ウエブ4を展綿する方が好ましく、また繊維シート3の両面に合繊ウエブを展綿しても実益は大きくない。

基布2にパイルを形成するには、該基布を裏返しにして公知のタフト機（図示しない）に送り、該タフト機によつてパイル糸5を刺し込む。タフト機でつくるパイルは、カツトパイル5a（第1図）でもループパイル5b（第2図）でもよく、または両者の混合パイルであつてもパイルが長いシヤギーであつてもよい。パイル糸5の素材としては、ナイロン、アクリル、ポリエステル、レーヨン、羊毛またはこれらの混紡糸などが例示できる。ついで基布2の裏面に適宜の接着剤6を塗布し、乾燥室に入れて高温で乾燥するとパイル5a，5bを基布2に保持する。接着剤には、一般にSBRラテックス、PVCペースト、酢酸ビニルラテックス、EVAラテックスなどをベースとするコンパウンドである。得たカーペット長尺物にブラッシングなどの後処理を施し、一定サイズに切断してから、オーバーロックミシンで耳かがり7（第4図参照）をすればよい。

また第3図に示すようなシヤギーボーダー加工を行なうには、カーペット長尺物を一定サイズに切断した後に、ミシンフック機（図示しない）で別のパイル糸を刺し込んで、長パイル8を形成させる。このシヤギーボーダー加工はオーバータフト加工ともいう。長パイル8はパイル5と着色が異なつていてもよく、種々の模様に刺し込むことができるけれども、通常ではカーペット周囲に帯状に配置させる。長パイル8の刺込み部分には、基布2の裏面において前記と同様の接着剤9をさらに塗布ついで乾燥して、長パイル8を基布2に固着させる。

この考案のカーペットを電熱カーペットのカバー材として用いるには、カーペット長尺物の切断または前記のシヤギーボーダー加工の後に、一般に不織布製である細長い台形布11（第4図）を4枚用意する。ついで基布裏面の各隅部において、台形布11の両側辺がカバー材周辺と合致するように配置してから、耳かがり7によつて同時に各台形布11を縫着する。この結果として、各台形布11の両側辺だけがカバー材10に縫着されるので、電熱カーペット12（第4図の一点鎖線参照）の各隅部を台形布11の下に差込むと、カバー材10の下側で電熱カーペット12を保留することができる。このような布11は、台形の代りに三角形であつてもよく、あるいはカバー材周辺の両長辺と一短辺に縫着するT字形布を2枚使用してもよい。

この考案に係るタフテツドカーペットは、繊維シートに合繊ウエブを一体化させた基布1枚を用いることにより、台地自体は従来よりも相当薄くても、カーペットの引裂強度およびパイル抜糸強度などの機械的性質がすぐれ、カーペットの重さや形態的安定性などの点でも好ましい。特にミシンフック機でカーペットにアクセントとしてシヤギーボーダー加工を施して、該カーペットの外観と触感を改善して商品価値を高める際に有利である。この考案のカーペットを電熱カーペットまたはそのカバー材として用いると、台地を単層の基布で構成するので薄く、下側に配置した面状ヒータからの発熱を上方へ良く伝え、電熱カーペット全体として熱効率がすぐれている。そして電熱カーペット用カバー材として、耳かがりの際に同時に台形布を基布裏面に縫着しても、該台形布を各隅部において堅固に取付けることができ、使用中に台形布が脱離することはほとんど生じない。またこの考案のカーペットをラグカーペットとして用いると、全体的に薄くなつて軽くかつ比較的曲げやすくなるので、カーペットの運搬や保管の際に有利になる。

次にこの考案を実施例によつて説明する。

実施例　1

電熱カーペット用カバー材の製造を目的として、ポリプロピレンのスリットヤーンの平織布（商品名ポリバック）の裏面に、6デニールで繊維長76㎜のポリエステル繊維100%からなる合繊

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ウエブ100g／㎡をニードルパンチングで一体化して基布2を得る。パイル糸として1500デニールの双糸撚のナイロンBCF糸を用い、タフテイング条件はゲージ1/8インチ、ステツチ6㎜、パイル長15㎜である。カットパイルのタフテイングの後に、基布裏面にEVAコンパウンドを固形分で900g／㎡塗布し、高温で乾燥する。得たカーペット長尺物を1000×2000㎜の長方形に切断する。

次にミシンフツクのためにパイル糸としてアクリル糸を用い、パイル本数10列、ゲージ1/8インチ、ステツチ5㎜、パイル長25㎜の条件によつて、カバー材周囲に帯状にシヤギーボーダー加工を施す。このパイル刺込み部分の基布裏面に、さらにEVAコンパウンドを固形部で150g／㎡塗布して乾燥する。本体と同じパイル糸による耳かがり7の際に、ポリエステル不織布の台形布11を各隅部に同時に縫付けて、電熱カーペット用カバー材10を得る。

比較例　1

平織布（商品名ポリバツク）に直接ナイロンBCF糸をタフテイングし、さらにミシンフツク機でシヤギーボーダー加工する。

比較例　2

ポリプロピレンのスパンボンドで不織布（商品名タイパー、デユポン社製）に直接ナイロンBCF糸をタフテイングし、さらにミシンフツク機でシヤギーボーダー加工する。

耳かがりの際に同時に縫着した台形布の縫製強度を調べると、5㎝巾の台形布で実施例1では15㎏、比較例1では4㎏、比較例2では5㎏である。またミシンフツク部分のパイル抜糸強度は、パイル1本あたり実施例1で3㎏、比較例1で1㎏、比較例2で0.5㎏である。したがつてカーペットの機械的性質に関して、実施例1のカバー材は比較例1および2のそれよりもすぐれている。

実施例　2

6

ラグカーペットの製造を目的として、3.7オンス／平方ヤードの不織布（商品名タイパー）の裏面に、6デニールで繊維長64㎜のポリプロピレン繊維100%からなる合繊ウエブ80g／㎡をニードルパンチングで一体化して基布2を得る。パイル糸として1050デニールの双方撚のナイロンBCF糸を用い、タフテイング条件はゲージ1/10インチ、ステツチ4.5㎜、パイル長7㎜である。ループパイルのタフテイングの後に、基布裏面にSBRコンパウンドを固形分で900g／㎡塗布し、高温で乾燥する。得たカーペット長尺物を950×1850㎜の長方形に切断する。

次にミシンフツクのためにパイル糸としてアクリル糸を用い、パイル本数8列、ゲージ1/8インチ、ステツチ5㎜、パイル長25㎜の条件によつて、カーペット周囲に帯状にシヤギーボーダー加工を施す。このパイル刺込み部分の基布裏面に、さらにEVAコンパウンドを固形分で150g／㎡塗布して乾燥する。さらに本体と同じパイル糸で耳かがりして、ラグカーペットを得る。

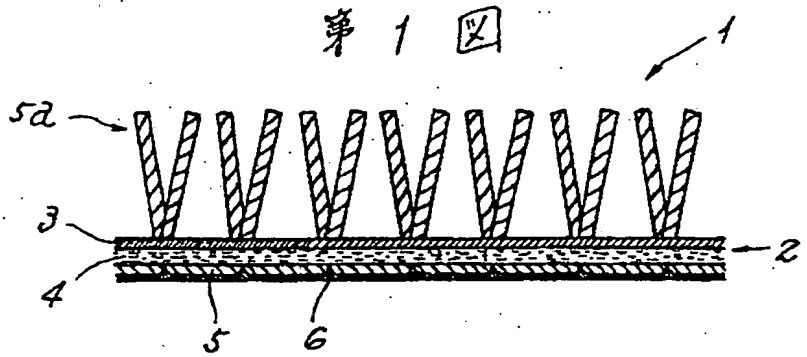
ミシンフツク部分のパイル抜糸強度は、実施例2では3㎏／本であるから、前記の比較例1および2の抜糸強度よりも大きく、機械的性質に関してすぐれている。

図面の簡単な説明

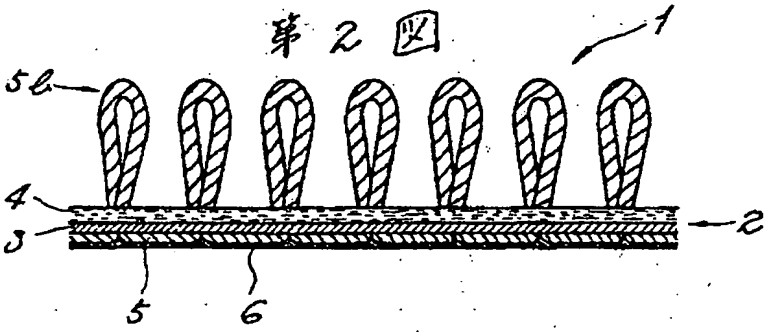
第1図および第2図はこの考案に係るタフテツドカーペットをそれぞれ例示する部分断面図、第3図はミシンフツク機によるシヤギーボーダー加工した後のカーペットを示す側面図、第4図はこの考案の一例である電熱カーペット用カバー材の裏面図である。

1……タフテツドカーペット、2……基布、3……繊維シート、4……合繊ウエブ、5……パイル糸、6……接着剤、7……耳かがり、8……長パイル、10……電熱カーペット用カバー材、11……台形布。

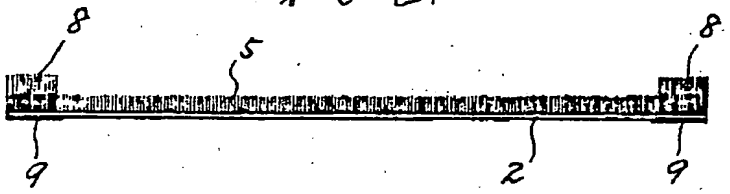
第 1 図



第 2 図



第 3 図



第 4 図